



(11) **EP 2 242 823 B1**

(12) **EUROPEAN PATENT SPECIFICATION**

(45) Date of publication and mention
of the grant of the patent:
13.03.2013 Bulletin 2013/11

(51) Int Cl.:
C10M 133/40 ^(2006.01) **C10M 169/06** ^(2006.01)
C10M 177/00 ^(2006.01)

(21) Application number: **09701564.8**

(86) International application number:
PCT/EP2009/050483

(22) Date of filing: **16.01.2009**

(87) International publication number:
WO 2009/090238 (23.07.2009 Gazette 2009/30)

(54) **METHOD FOR PREPARING A GREASE COMPOSITION**

VERFAHREN ZUR HERSTELLUNG EINER SCHMIERFETTZUSAMMENSETZUNG

PROCÉDÉ DE PRÉPARATION D'UNE COMPOSITION DE GRAISSE

(84) Designated Contracting States:
**AT BE BG CH CY CZ DE DK EE ES FI FR GB GR
HR HU IE IS IT LI LT LU LV MC MK MT NL NO PL
PT RO SE SI SK TR**

(30) Priority: **16.01.2008 EP 08100550**

(43) Date of publication of application:
27.10.2010 Bulletin 2010/43

(73) Proprietor: **Shell Internationale Research
Maatschappij B.V.
2596 HR The Hague (NL)**

(72) Inventor: **WHEATLEY, Alan Richard
Ince,
Chester,
Cheshire CH2 4NU (GB)**

(74) Representative: **Matthezing, Robert Maarten
Shell International B.V.
Intellectual Property Services
P.O. Box 384
2501 CJ The Hague (NL)**

(56) References cited:
**GB-A- 808 596 US-A- 2 908 646
US-A- 5 246 606 US-A1- 2006 205 613**

- **CASERI W: "Polyalkylenglycole", INTERNET CITATION, 1 January 2010 (2010-01-01), pages 1-2, XP007916093, Retrieved from the Internet: URL: <http://www.roempp.com/prod/roempp.php> [retrieved on 2010-12-03]**
- **CLARIANT: "Ethylene glycols, mono and dialkyl glycol ethers - Ethylenglykole, Mono- und Diälylglykoether", PRODUCT RANGE CLARIANT, CLAIRANT, DE, 1 April 2000 (2000-04-01), pages 1-24, XP007916094,**

Note: Within nine months of the publication of the mention of the grant of the European patent in the European Patent Bulletin, any person may give notice to the European Patent Office of opposition to that patent, in accordance with the Implementing Regulations. Notice of opposition shall not be deemed to have been filed until the opposition fee has been paid. (Art. 99(1) European Patent Convention).

EP 2 242 823 B1

Description

[0001] The present invention relates to a method for preparing a lubricating composition, in particular a grease.

[0002] Alkyl-substituted quinolines and polymerized derivatives thereof are known as very effective, low cost antioxidants for several uses, including the use in lubricating compositions.

[0003] As an example, WO 94/24235 discloses the use of alkyl-substituted 1,2-dihydroquinolines (including monomers, dimers, trimers and tetramers thereof) in motor oils, transmission oils, gear oils, metal working fluids, hydraulic fluids, greases and the like. Illustrative examples of these alkyl-substituted 1,2-dihydroquinolines are 2,2,4-trimethyl-1,2-dihydroquinoline, 2-methyl-2,4-diethyl-1,2-dihydroquinoline, 2,2,4,6-tetramethyl-1,2-dihydroquinoline, 2,2,4,7-tetramethyl-1,2-dihydroquinoline, 6,6'-bis(2,2,4-trimethyl-1,2-dihydroquinoline) and the like.

[0004] Further, US 5 246 606 discloses that dimeric, trimeric and tetrameric tetrahydroquinoline derivatives are suitable for stabilising organic materials against light-induced, thermal and/or oxidative degradation. US 5 246 606 suggests to use these compounds amongst others in functional fluids such as lubricants and hydraulic fluids.

[0005] US 2 908 646 relates to lubricating greases containing polymerized dihydroquinoline derivatives. This document teaches that the polymerized dihydroquinoline derivatives may be added directly to the grease mixture or may be added in the form of a solution. In the example of US 2 908 646, a polymerized 2,2,4-trimethyl-1,2-dihydroquinoline is added during the oil addition at a temperature of 240°F.

[0006] Although alkyl-substituted quinolines such as 2,2,4-trimethyl-1,2-dihydroquinoline (also referred to as "TMQ", "TMDQ" and "TMHQ") and oligomeric (i.e. dimeric, trimeric and tetrameric) derivatives thereof, are widely used as antioxidants, they have a number of disadvantages.

[0007] As an example, TMQ is in the form of a brittle solid at room temperature and, being usually in the form of a mixture of monomer and oligomers, it has no well-defined melting point. Although it softens as the temperature increases, it is still a very viscous and stringy material at typical additive addition temperatures of around 80 to 100°C.

[0008] If TMQ is added to lubricating compositions such as greases at this typical temperature range of from 80 to 100°C, it will not be suitably dispersed into the grease and will result in e.g. filter blockages in grease delivery systems. Even if the filter loading of these grease delivery systems is not enough for the filter to block immediately, some of the antioxidant will have been taken out of the grease, which would then suffer from reduced life as a result.

[0009] In view of the above, alkyl-substituted quinolines are usually added to greases at temperatures above 150°C, usually between 150-160°C after the grease has completed its critical cooling phase after establishing the thickener system in the base oil.

[0010] However, an associated problem of the known method is that the window of opportunity for adding the alkyl-substituted quinolines is usually narrow, if they are to be dispersed properly in the grease. If the alkyl-substituted quinolines are added e.g. ten minutes later, this may be too late as the grease may have been cooled too much.

[0011] A further problem of the known method of adding the alkyl-substituted quinolines to the grease at relatively high temperatures, is that more severe health and safety issues need to be taken into account, relating e.g. to the dangers of grease at high temperature and the danger of fumes when the manufacturing vessel has to be opened.

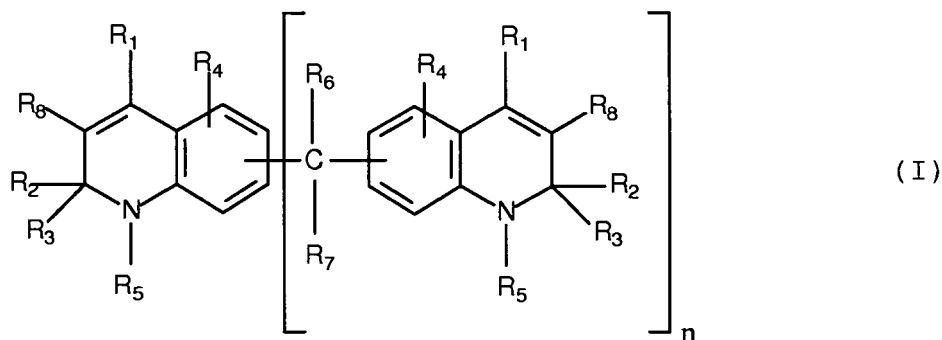
[0012] It is an object of the present invention to avoid the above problems.

[0013] It is another object to provide an alternative method for preparing a lubricating composition, in particular a grease.

[0014] One or more of the above or other objects are obtained by the present invention by providing a method for preparing a lubricating composition, in particular a grease, the method at least comprising the steps of:

a) providing a base oil composition containing one or more additives, wherein the base oil composition comprises mineral oil;

b) providing a solution of one or more alkyl-substituted quinolines in a solvent, wherein the one or more alkyl-substituted quinolines are alkyl-substituted 1,2-dihydroquinolines having the general formula (I)



wherein R^1 - R^8 are independently selected from hydrogen or an alkyl group having 1-8 carbon atoms; and wherein the alkyl-substituted 1,2-dihydroquinolines have an average value for n of from 1.0 to 2.0 and wherein the solvent comprises a polyalkylene glycol having alkylene oxide units with 1 to 6 carbon atoms as monomer units, and wherein the polyalkylene glycol has a kinematic viscosity of 10 to 400 mm²/s measured at 40°C according to DIN 51562; and

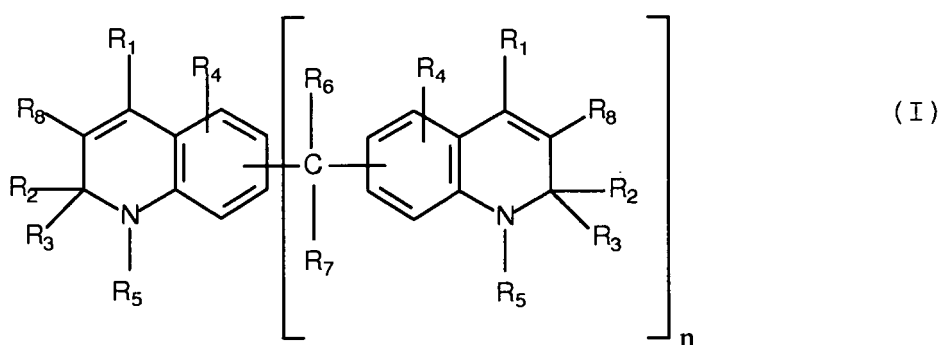
c) adding the solution of step b) to the base oil composition of step a) at a temperature below 150°C.

[0015] Surprisingly, it has been found according to the present invention that if the alkyl-substituted quinolines (or oligomeric derivatives thereof) are added in a solvent to the base oil composition, they can be added at a lower temperature, whilst still obtaining a proper dispersing thereof in the base oil composition.

[0016] An important advantage of the present invention is that the alkyl-substituted quinolines (or oligomeric derivatives thereof) can be added at a lower temperature, resulting in less severe safety requirements. Also, there is more flexibility in the moment of adding the alkyl-substituted quinolines (or oligomeric derivatives thereof), as the specific temperature for adding thereof is less critical than in the case where no solvent is used.

[0017] Preferably in step c) the solution of step b) is added at a temperature below 120°C, preferably in the range of from 10 to 110°C, more preferably from 15 to 100°C.

[0018] According to the present invention, the one or more alkyl-substituted quinolines are alkyl-substituted 1,2-dihydroquinolines having the general formula (I)



wherein R^1 - R^8 are independently selected from hydrogen or an alkyl group having 1-8 carbon atoms.

[0019] Preferably, R^1 - R^8 are independently selected from hydrogen or an alkyl group having 1-4 carbon atoms, preferably having 1-2 carbon atoms. Preferably R^4 is H. It is even more preferred that R^4 - R^8 are all H. Also it is preferred that R^1 - R^3 are all a methyl group.

[0020] Further the one or more alkyl substituted 1,2-dihydroquinolines have an average value for n of from 1.0 to 2.0, preferably from 1.3 to 1.6.

[0021] Also it is preferred that the one or more alkyl substituted quinolines provided in the solution have a solubility of below 0.1% as determined using ASTM D893.

[0022] The alkyl substituted quinoline compounds as used in the present invention are either commercially available or can be prepared by various reactions that are known in the art. Examples of preparation methods have been given in the above-mentioned WO 94/24235 and US 5 246 606 and references cited therein. Other examples are given in US 4 692 258 and US 3 910 918 and references cited therein.

[0023] One specific example for preparing TMQ (2,2,4-trimethyl-1,2-dihydroquinoline) has been given in W.R. Vaughan, "Organic synthesis", Collective Volume III, pp 329-30, (1955).

[0024] The solvent comprises a polyalkylene glycol. Polyglycols are well known in the art and are not further discussed here in detail.

[0025] The polyalkylene glycols (PAG) exhibit alkylene oxide units with 1 to 6 carbon atoms (-R-O-) as monomer units.

[0026] The polyalkylene glycols may exhibit hydrogen end groups, alkyl, aryl, alkylaryl, aryloxy, alkoxy, alkylaryloxy and/or hydroxy end groups. Alkylaryloxy groups should also be understood to mean arylalkyl (ene)oxy groups and alkylaryl groups to mean arylalkyl(ene) groups (e.g. aryl CH₂CH₂-). The end groups of the alkyl type, including the alkoxy type, or of the aryl types, including the alkylaryl type, aryloxy type and alkylaryloxy type preferably exhibit 6 to 24 carbon atoms, particularly preferably 6 to 18 carbon atoms, based on the aryl types, and preferably 1 to 12 carbon atoms, based on the alkyl types.

[0027] The polyalkylene glycols according to the invention may be either homopolymers, namely polypropylene glycol (and/or polypropylene oxide) or copolymers, terpolymers etc. For the latter cases, the monomer units may exhibit a random distribution or a block structure. If the polyalkylene glycols are not homopolymers, preferably at least 20%, preferably at least 40% of all monomer units are producible from polypropylene oxide (PO), and also preferably, at least 20% of all monomer units of these polyalkylene glycols are producible by using ethylene oxide (EO) (PO/EO copolymers).

According to a further embodiment, preferably at least 20%, preferably at least 40% of all monomer units are obtainable from butylene oxide (BO) and, moreover, preferably at least 20% of all monomer units of these polyalkylene glycols are obtainable by using ethylene oxide (BO/EO copolymers).

[0028] When (poly)alcohols are used, the starting compound is incorporated into the polymer and, according to the meaning of the invention, also referred to as end group of the polymer chain. Suitable starting groups consist of compounds comprising active hydrogen such as e.g. n-butanol, propylene glycol, ethylene glycol, neopentyl glycols such as pentaerythritol, ethylene diamine, phenol, cresol or other (C1 to C16 (mono, di or tri)alkyl) aromatics, (hydroxyalkyl) aromatics, hydroquinone, aminoethanolamines, triethylenetetramines, polyamines, sorbitol or other sugars. Other C-H acidic compounds such as carboxylic acids or carboxylic anhydrides can also be used as starting compounds.

[0029] Preferably, the polyalkylene glycols comprise aryl groups or corresponding heteroaromatic groups, e.g. inserted into the polymer chain, as side groups or end groups; the groups may, if necessary, be substituted with linear or branched alkyl groups or alkylene groups, the alkyl groups or alkylene groups overall exhibiting preferably 1 to 18 carbon atoms.

[0030] Cyclic ether alcohols such as hydroxyfurfuryl or hydroxytetrahydrofuran, nitrogen heterocyclics or sulphur heterocyclics can also be used as starting groups. Such polyalkylene glycols are disclosed in WO 01/57164, the teaching of which is herewith incorporated by reference.

[0031] Preferably, the polyalkylene glycols according to the invention have an average molecular weight (number average) of 200 to 3000 g/mole, more preferably 400 to 2000 g/mole. The kinematic viscosity of the polyalkylene glycols is 10 to 400 mm²/s (cSt) measured at 40°C according to DIN 51562.

[0032] The polyalkylene glycols used according to the invention can be produced by reacting alcohols, including polyalcohols, as starting compounds with oxiranes such as ethylene oxide, propylene oxide and/or butylene oxide. Following the reaction, these possess only one free hydroxy group as end group. Polyalkylene glycols with only one hydroxy group are preferred over those with two free hydroxy groups. Polyalkylene glycols which, e.g. after a further etherification step, comprise no free hydroxy groups any longer are particularly preferred regarding the stability, hygroscopicity and compatibility. The alkylation of terminal hydroxyl groups leads to an increase in the thermal stability. Thus, in an especially preferred embodiment according to the present invention, the PAG base oil comprises end-capped PAG, i.e. where no free hydroxyl groups are present.

[0033] In the base oil composition used in the method according to the present invention various conventional mineral oils and synthetic oils may be conveniently used. For the purpose of this description, the term "base oil" is meant to also include a grease base stock.

[0034] The base oil composition used in the present invention may conveniently comprise mixtures of one or more mineral oils and/or one or more synthetic oils.

[0035] Mineral oils include liquid petroleum oils and solvent-treated or acid-treated mineral lubricating oil of the paraffinic, naphthenic, or mixed paraffinic/naphthenic type which may be further refined by hydrofinishing processes and/or dewaxing.

[0036] Suitable base oils for use in the lubricating oil composition of the present invention are Group I, Group II or Group III base oils, polyalphaolefins, Fischer-Tropsch derived base oils and mixtures thereof.

[0037] By "Group I" base oil, "Group II" base oil and "Group III" base oil in the present invention are meant lubricating oil base oils according to the definitions of American Petroleum Institute (API) categories I, II and III. Such API categories are defined in API Publication 1509, 15th Edition, Appendix E, April 2002.

[0038] Suitable Fischer-Tropsch derived base oils that may be conveniently used in the base oil in the lubricating oil composition of the present invention are those as for example disclosed in EP 0 776 959, EP 0 668 342, WO 97/21788, WO 00/15736, WO 00/14188, WO 00/14187, WO 00/14183, WO 00/14179, WO 00/08115, WO 99/41332, EP 1 029 029, WO 01/18156 and WO 01/57166.

[0039] Synthetic oils include hydrocarbon oils such as olefin oligomers (PAOs), dibasic acid esters, polyol esters, and dewaxed waxy raffinate. Synthetic hydrocarbon base oils sold by the Shell Group under the designation "XHVI" (trade mark) may be conveniently used.

[0040] The total amount of base oil incorporated in the lubricating composition of the present invention is preferably present in an amount in the range of from 60 to 92 wt.%, more preferably in an amount in the range of from 75 to 90 wt.% and most preferably in an amount in the range of from 75 to 88 wt.%, with respect to the total weight of the lubricating composition.

[0041] If desired, the final lubricating composition may further comprise one or more additives such as antioxidants, anti-wear agents, dispersants, detergents, friction modifiers, viscosity index improvers, pour point depressants, tackifying agents, corrosion inhibitors, demulsifiers, defoaming agents and seal fix or seal compatibility agents, etc.

[0042] As the person skilled in the art is familiar with the above and other additives, these are not further discussed here. The additives may be added to the base oil composition before or after the one or more alkyl-substituted quinolines are added in step c). Also, if appropriate, the additives may also be added at the same time with the one or more alkyl-substituted quinolines.

[0043] Said additives are typically present in an amount in the range of from 0.01 to 12.5 wt.%, based on the total

weight of the lubricating composition, preferably in an amount in the range of from 0.05 to 10.0 wt.%, more preferably from 1.0 to 9.0 wt.% and most preferably in the range of from 2.0 to 5.0 wt.%, based on the total weight of the lubricating composition.

[0044] As the lubricating composition may also be (and preferably is) in the form of a grease, the base oil as contained in the lubricating composition may contain or be compounded with one or more thickeners such as metallic soaps, organic substances or inorganic substances, for example, lithium soaps, lithium complex soaps, sodium terephthalate, urea/urethane compounds and clays.

[0045] Preferably, the lubricating composition has a kinematic viscosity in the range of from 2 to 80 mm²/s at 100 °C, more preferably in the range of from 3 to 70 mm²/s, most preferably in the range of from 4 to 50 mm²/s.

[0046] The lubricating compositions of the present invention may be conveniently prepared by admixing the one or more base oils and, optionally, one or more additives that are usually present in lubricating compositions, for example as herein before described, with mineral and/or synthetic base oil. Preferably, and as is customary in the art, the one or more alkyl-substituted quinoline compounds (or oligomeric derivatives thereof) have a sufficiently small particle size (e.g. below 50 µm, preferably below 20 µm) to allow easy dispersion thereof in the lubricating composition.

[0047] In another aspect the present invention provides a lubricating composition, in particular a grease, obtained by the method according to the present invention.

[0048] The present invention is described below with reference to the following Examples, which are not intended to limit the scope of the present invention in any way.

Examples

Solutions of Alkyl-Substituted Quinolines

- Solution A

[0049] A 500 ml 50 % m/m solution of oligomeric 2,2,4-trimethyl-1,2-dihydroquinoline (solid; available from Rhein Chemie Rheinau GmbH under the trade designation "Additin RC7010") in polyalkylene glycol (available from The Dow Chemical Company, USA under the trade designation "Oxilube 504") was prepared by heating the polyalkylene glycol to 100°C before adding the oligomeric 2,2,4-trimethyl-1,2-dihydroquinoline. The oligomeric 2,2,4-trimethyl-1,2-dihydroquinoline was added slowly over about two minutes, allowing it to disperse throughout the fluid before adding more. The mixture thus obtained was stirred for a further 30 minutes at 100°C. A stable, homogeneous solution was formed.

- Solution B

[0050] Similar to Solution A, a 500 ml 50% m/m solution of oligomeric 2,2,4-trimethyl-1,2-dihydroquinoline in polyglycol (available from The Dow Chemical Company, USA under the trade designation "Synalox 50-50B") was prepared. A stable, homogeneous solution was formed.

Example 1

[0051] A grease was prepared using the above mentioned Solution A (50 ml) and a conventional grease base stock (4950 g).

[0052] The conventional grease base stock contained about 10% m/m lithium complex thickener and about 90% m/m paraffinic mineral base oil blended from SN 500 and bright stock (viscosity at 40°C of 180 mm²/s according to ASTM D445). The conventional grease base stock also contained an antiwear additive (a zinc dialkyl dithiophosphate), an extreme pressure additive (a sulphurised ester) and a rust inhibitor (a zinc naphthenate).

[0053] The grease base stock and Solution A were simply mixed during 30 minutes using a laboratory paddle mixer, after both had been previously heated to 80°C before mixing.

[0054] After visual inspection no apparent deficiencies of the obtained grease were found. No lumps were formed. Also, no separating of the oligomeric 2,2,4-trimethyl-1,2-dihydroquinoline out of the grease occurred after 12 months of storage.

[0055] Other properties of the obtained grease are indicated in Table I below.

Table I

Test	Method / Conditions	Value
Penetration (worked)	ASTM D217 25°C	260

(continued)

Test	Method / Conditions	Value
Penetration (long term worked)	ASTM D217 100,000 strokes	280
Oil separation	IP 121 7 days at 40°C	3.5%
Oxidation stability	ASTM D942 100h at 99°C	30 kPa
Copper corrosion	ASTM D4048 24h at 100°C	1b
Water washout	ASTM D1264 1h at 79°C	0.5%

[0056] The results in Table I show that the desired properties for a grease such as mechanical stability, oil retention and surprisingly also the stability against water are still present in the grease of Example 1 and are not affected by the presence of the solvent (polyalkylene glycol) used to dissolve the oligomeric 2,2,4-trimethyl-1,2-dihydroquinoline.

Example 2

[0057] Similar to Example 1, a grease was prepared using Solution B.

[0058] After visual inspection no apparent deficiencies of the obtained grease were found. No lumps were formed.

[0059] Also, no separating of the oligomeric 2,2,4-trimethyl-1,2-dihydroquinoline 2,2,4-trimethyl-1,2-dihydroquinoline out of the grease occurred after 12 months of storage. Similar to Example 1, the grease of Example 2 showed desired properties of mechanical stability, oil retention and stability against water.

Comparative Example 1

[0060] A grease was prepared using the same amount of oligomeric 2,2,4-trimethyl-1,2-dihydroquinoline (without the solvent) and the same grease base stock of Examples 1 and 2.

[0061] The grease base stock was heated to about 160°C. Then, a part of the solid oligomeric 2,2,4-trimethyl-1,2-dihydroquinoline was added to the heated grease base stock and left at this temperature for about 10 minutes. Mixing took place using a laboratory paddle mixer until the mixture had reached the temperature of about 60°C.

[0062] Then the mixture was reheated to about 160°C and left at this temperature for about 30 minutes. Subsequently, the steps of adding and mixing was repeated until all oligomeric 2,2,4-trimethyl-1,2-dihydroquinoline was added and mixed.

[0063] After visual inspection no apparent deficiencies of the obtained grease were found. No lumps were formed.

Comparative Example 2

[0064] Similar to Comparative Example 1 a grease was prepared, although the heating was to about 60°C (instead of to about 160°C).

[0065] It was not possible to disperse the solid oligomeric 2,2,4-trimethyl-1,2-dihydroquinoline into the grease base stock, which would lead to severe filter blockages in case the grease of Comparative Example 2 was to be used in e.g. grease delivery systems.

Discussion

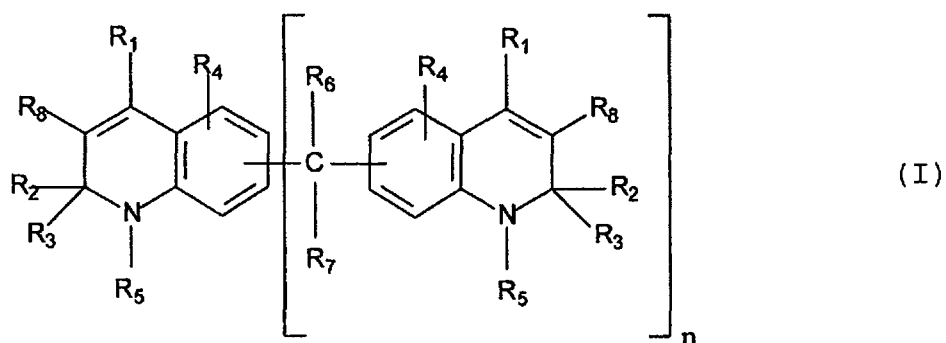
[0066] As can be seen from the Examples, the present invention allows adding the alkyl-substituted quinolines or oligomeric derivatives thereof at a significant lower temperature, whilst still obtaining a stable grease having desired properties. It goes without saying that this is highly desired from a health and safety perspective as well as from a practical manufacturing perspective.

[0067] In this respect it is noted that if no solvent was used (see Comparative Example 2) whilst adding the alkyl-substituted quinolines or oligomeric derivatives thereof, no stable grease was obtained.

Claims

1. A method for preparing a grease, the method at least comprising the steps of:

- a) providing a base oil composition containing one or more additives, wherein the base oil composition comprises mineral oil;
- b) providing a solution of one or more alkyl-substituted quinolines in a solvent, wherein the one or more alkyl-substituted quinolines are alkyl-substituted 1,2-dihydroquinolines having the general formula (I)

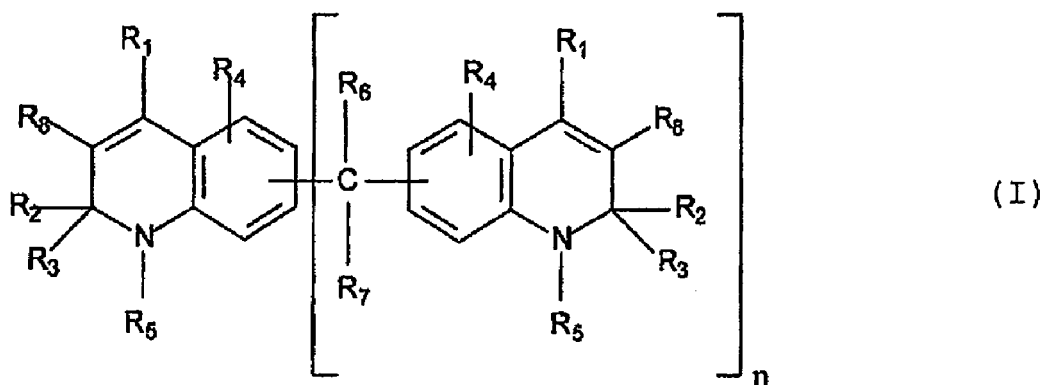


- wherein R^1 - R^8 are independently selected from hydrogen or an alkyl group having 1-8 carbon atoms; and wherein the alkyl-substituted 1,2-dihydroquinolines have an average value for n of from 1.0 to 2.0 and wherein the solvent comprises a polyalkylene glycol having alkylene oxide units with 1 to 6 carbon atoms as monomer units, and wherein the polyalkylene glycol has a kinematic viscosity of 10 to 400 mm²/s measured at 40°C according to DIN 51562; and
- c) adding the solution of step b) to the base oil composition of step a) at a temperature in the range of from 10 to 110°C.
2. The method according to claim 1, wherein in step c) the solution of step b) is added at a temperature in the range of from 15 to 100°C.
 3. The method according to claim 1 or 2, wherein R^1 - R^8 are independently selected from hydrogen or an alkyl group having 1-4 carbon atoms.
 4. The method according to any of claims 1 to 3, wherein R^1 - R^8 are independently selected from hydrogen or an alkyl group having 1-2 carbon atoms.
 5. The method according to any of claims 1 to 4, wherein R^4 - R^8 are hydrogen.
 6. The method according to any of claims 1 to 5, wherein the one or more alkyl-substituted 1,2-dihydroquinolines have an average value for n of from 1.3 to 1.6.
 7. The method according to any one of claims 1 to 6, wherein the one or more alkyl substituted quinolines provided in the solution have a solubility in the solvent of below 0.1% as determined using ASTM D893.

Patentansprüche

1. Verfahren zur Herstellung eines Fettes, welches Verfahren mindestens die folgenden Schritte umfasst:

- a) Bereitstellen einer Basisölszusammensetzung, welche eines oder mehrere Additive enthält, wobei die Basisölszusammensetzung Mineralöl umfasst;
- b) Bereitstellen einer Lösung eines oder mehrerer alkylsubstituierter Chinoline in einem Lösungsmittel, wobei das eine oder die mehreren alkylsubstituierten Chinoline alkylsubstituierte 1,2-Dihydrochinoline mit der allgemeinen Formel (I)

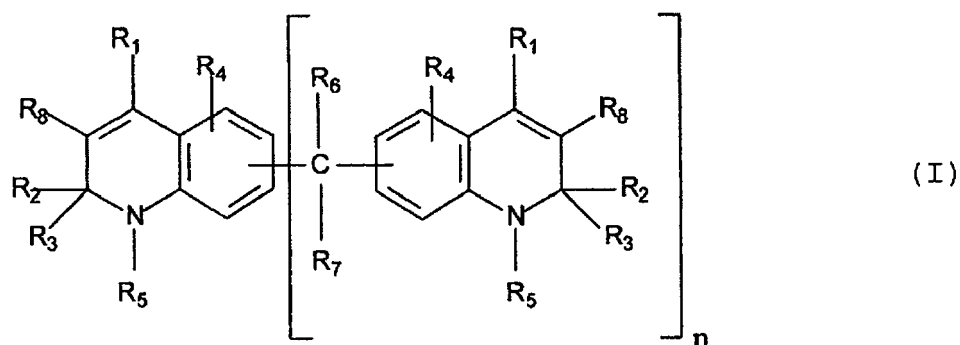


sind,
wobei R¹ - R⁸ unabhängig voneinander wahlweise Wasserstoff oder eine Alkylgruppe mit 1 - 8 Kohlenstoffatomen
sind;
und wobei die alkylsubstituierten 1,2-Dihydrochinoline einen durchschnittlichen Wert für n von 1,0 bis 2,0 auf-
weisen und wobei das Lösungsmittel ein Polyalkylenglykol mit Alkylendioxid-Einheiten mit 1 bis 6 Kohlenstoffa-
tomen als Monomer-Einheiten umfasst, und wobei das Polyalkylenglykol eine kinematische Viskosität von 10
bis 400 mm²/s aufweist, gemessen bei 40 °C gemäß DIN 51562; und
c) Zugabe der Lösung aus Schritt b) zu der Basisölszusammensetzung aus Schritt a) bei einer Temperatur im
Bereich von 10 bis 110 °C.

2. Verfahren nach Anspruch 1,
wobei in Schritt c) die Lösung aus Schritt b) bei einer Temperatur im Bereich von 15 bis 100 °C zugegeben wird.
3. Verfahren nach Anspruch 1 oder 2,
wobei R¹ - R⁸ unabhängig voneinander wahlweise Wasserstoff oder eine Alkylgruppe mit 1 - 4 Kohlenstoffatomen
sind.
4. Verfahren nach einem der Ansprüche 1 bis 3,
wobei R¹ - R⁸ unabhängig voneinander wahlweise Wasserstoff oder eine Alkylgruppe mit 1 - 2 Kohlenstoffatomen
sind.
5. Verfahren nach einem der Ansprüche 1 bis 4,
wobei R⁴ - R⁸ Wasserstoff sind.
6. Verfahren nach einem der Ansprüche 1 bis 5,
wobei das eine oder die mehreren alkylsubstituierten 1,2-Dihydrochinoline einen durchschnittlichen Wert für n von
1,3 bis 1,6 aufweisen.
7. Verfahren nach einem der Ansprüche 1 bis 6,
wobei das eine oder die mehreren in der Lösung bereitgestellten alkylsubstituierten Chinoline eine unter Anwendung
von ASTM D893 ermittelte Löslichkeit in dem Lösungsmittel von unter 0,1 % aufweisen.

Revendications

1. Procédé de préparation d'une graisse, le procédé comprenant au moins les étapes consistant à :
a) mettre en oeuvre une composition d'huile de base contenant un ou plusieurs additifs, dans lequel la compo-
sition d'huile de base comprend de l'huile minérale ;
b) mettre en oeuvre une solution d'une ou plusieurs quinoléines à substitution alkyle, dans lequel la ou les
quinoléines à substitution alkyle est ou sont des 1,2-dihydroquinoléines à substitution alkyle ayant la formule
générale (I) :



dans lequel procédé R¹ à R⁸ sont indépendamment choisis parmi l'hydrogène ou un groupement alkyle ayant 1 à 8 atomes de carbone ; dans lequel les 1,2-dihydroquinoléines à substitution alkyle ont une valeur moyenne pour n de 1,0 à 2,0, dans lequel le solvant comprend un polyalkylèneglycol ayant des unités oxyde d'alkylène avec 1 à 6 atomes de carbone comme unités monomères, et dans lequel le polyalkylèneglycol a une viscosité cinématique de 100 à 400 mm²/s mesurée à 40 °C selon la norme DIN 51562 ; et

c) ajouter la solution de l'étape b) à la composition d'huile de base de l'étape a) à une température dans la plage de 10 à 110 °C

2. Procédé selon la revendication 1, dans lequel, à l'étape c), la solution de l'étape b) est ajoutée à une température dans la plage de 15 à 100 °C.
3. Procédé selon la revendication 1 ou la revendication 2, dans lequel R¹ à R⁸ sont indépendamment choisis parmi l'hydrogène ou un groupement alkyle ayant 1 à 4 atomes de carbone.
4. Procédé selon l'une quelconque des revendications 1 à 3, dans lequel R¹ à R⁸ sont indépendamment choisis parmi l'hydrogène ou un groupement alkyle ayant 1 à 2 atomes de carbone.
5. Procédé selon l'une quelconque des revendications 1 à 4, dans lequel R⁴ à R⁸ sont de l'hydrogène.
6. Procédé selon l'une quelconque des revendications 1 à 5, dans lequel la ou les 1,2-dihydroquinoléines à substitution alkyle a ou ont une valeur moyenne pour n de 1,3 à 1,6.
7. Procédé selon l'une quelconque des revendications 1 à 6, dans lequel la ou les quinoléines à substitution alkyle mises en oeuvre dans la solution a ou ont une solubilité dans le solvant en dessous de 0,1 %, comme déterminé en utilisant la norme ASTM D893.

REFERENCES CITED IN THE DESCRIPTION

This list of references cited by the applicant is for the reader's convenience only. It does not form part of the European patent document. Even though great care has been taken in compiling the references, errors or omissions cannot be excluded and the EPO disclaims all liability in this regard.

Patent documents cited in the description

- | | |
|------------------------------|-----------------------|
| • WO 9424235 A [0003] [0022] | • WO 0014188 A [0038] |
| • US 5246606 A [0004] [0022] | • WO 0014187 A [0038] |
| • US 2908646 A [0005] | • WO 0014183 A [0038] |
| • US 4692258 A [0022] | • WO 0014179 A [0038] |
| • US 3910918 A [0022] | • WO 0008115 A [0038] |
| • WO 0157164 A [0030] | • WO 9941332 A [0038] |
| • EP 0776959 A [0038] | • EP 1029029 A [0038] |
| • EP 0668342 A [0038] | • WO 0118156 A [0038] |
| • WO 9721788 A [0038] | • WO 0157166 A [0038] |
| • WO 0015736 A [0038] | |

Non-patent literature cited in the description

- **W.R. VAUGHAN.** *Organic synthesis*, 1955, vol. III, 329-30 [0023]